

M.Sc. Project Report on
Synthesis and Characterization of YBCO/CNT
Composite

Submitted by
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CERTIFICATE

This is to certify that the project report titled “**Synthesis and Characterization of YBCO/CNT composite**” is submitted by **Ms Adyasha Aparimita** in partial fulfillment of the constraint towards the award of Master of Science degree in Physics at NIT, Rourkela. This is a bonafied work carried out by her under my administration and supervision in low temperature laboratory of the Department of Physics and Astronomy.

To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other University/Institution.

Prof. D. Behera
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ACKNOWLEDGEMENT

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ABSTRACT

YBCO are synthesized by solid state reaction method. From XRD data, the orthorhombic structure is confirmed and measurement of resistance versus temperature is taken by four probe method. YBCO is a well-studied superconducting material has a phase transition 92 K which is above the boiling point of liquid nitrogen. CNT was added to YBCO to form a composite in order to increase its current density and observe the changes in its properties which arises due to the mixing the CNT. And phase confirmation of YBCO composite is done using XRD and microstructural analysis is performed using SEM. The resistivity vs. temperature measurement is done by four probe method. Now a days, research is going on to find the mechanism of high temperature superconductivity material as BCS theory is silent about this.

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CHAPTER-1

INTRODUCTION:

Superconductivity was discovered by Dutch Physicist Heike KamerlinghOnnes in 1911 while he was working at low temperature. Superconductivity is the property of metal, alloys, and chemical compound at a certain temperature called transition temperature or critical temperature where electrical resistivity vanishes to zero and they become perfectly diamagnetic in nature. When we apply a magnetic field is applied to a superconductor, then these magnetic lines of force are expelled out from this material. This property of a superconductor is due to Meissner effect. A vast change in the research area has taken place when high temperature superconductors (HTSC) having T_c of 92 K which is above the boiling point of liquid nitrogen (77 K), was discovered in $YBa_2Cu_3O_{7-\delta}$ (YBCO) in the year 1986. This superconductor with the property of zero resistance at liquid-nitrogen temperature has a worldwide interest in power applications like superconducting magnets, motors and power-transmission lines etc. In the field of electronics, extensive studies on Josephson junction, Josephson computers and the progress of commercial superconducting quantum interference device (SQUID) systems are now applied for characterization of materials. This application of the superconductor is of most importance.

1.1Types of superconductors:

- Type - I superconductors (Low T_c superconductors)
- Type - II superconductors (High T_c superconductor)

1.1.1 Type-I superconductor:

The superconducting materials which exhibit total expulsion of flux (Meissner effect) up to a critical magnetic field (H_c) beyond this magnetic field flux penetrate the material and it behaves as normal conductor. This type of superconductors are called type-I superconductor. Pristine samples of Pb, Hg and Sn are examples of Type-I superconductors. These superconductors are also known as soft normal superconductors.

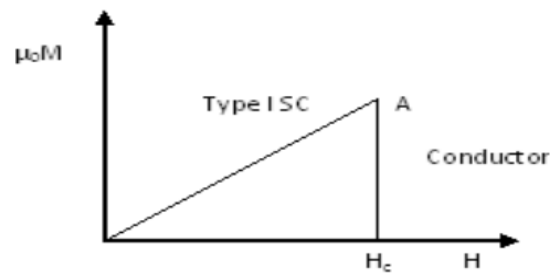


Figure-1

1.1.2 Type-II superconductors:

Type-II superconductors are also known as hard superconductors which can bear high magnetic field. High temperature superconductors like YBCO, BSCCO, TIBCCO, and HBCCO are the examples of Type II superconductors. This type of superconductivity is showed by a metal which attains high values of the electrical resistivity in the normal state for which mean free path is short. The figure below shows the behavior of magnetic field to material. It obeys Meissner effect up to certain critical field H_{c1} at which magnetic flux lines begins to enter the superconductor and at an upper critical field H_{c2} the superconductivity behavior disappears.

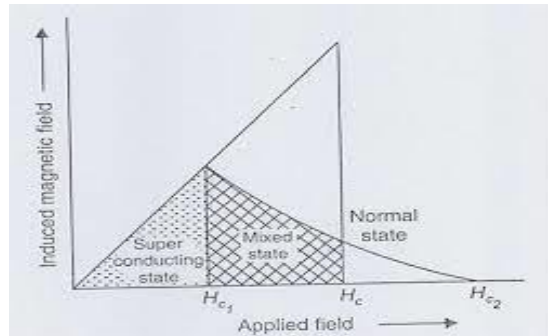


Figure-2

1.1.3 High- T_c superconductor:

High T_c or HTS denotes superconductivity in materials, chiefly oxides, with high temperatures, accompanied by high critical currents and magnetic fields. High temperature superconductors denote superconductivity with high transition temperature accompanied by high critical current and magnetic field. CuO_2 layer is the basic feature of all HTC with transition temperature greater than 40 K. More than 135 K can be achieved. Some examples are LBSO, YBCO, TBCCO, HBCCO etc.

1.2 Properties of superconductors:

Most of the physical properties of superconductors vary from material to material, for example the heat capacity and the critical temperature, critical current density, and critical field at which superconductivity is destroyed. For instance, all superconductors have exactly zero resistivity to low applied currents when there is no magnetic field present or if the applied field does not exceed a critical value.

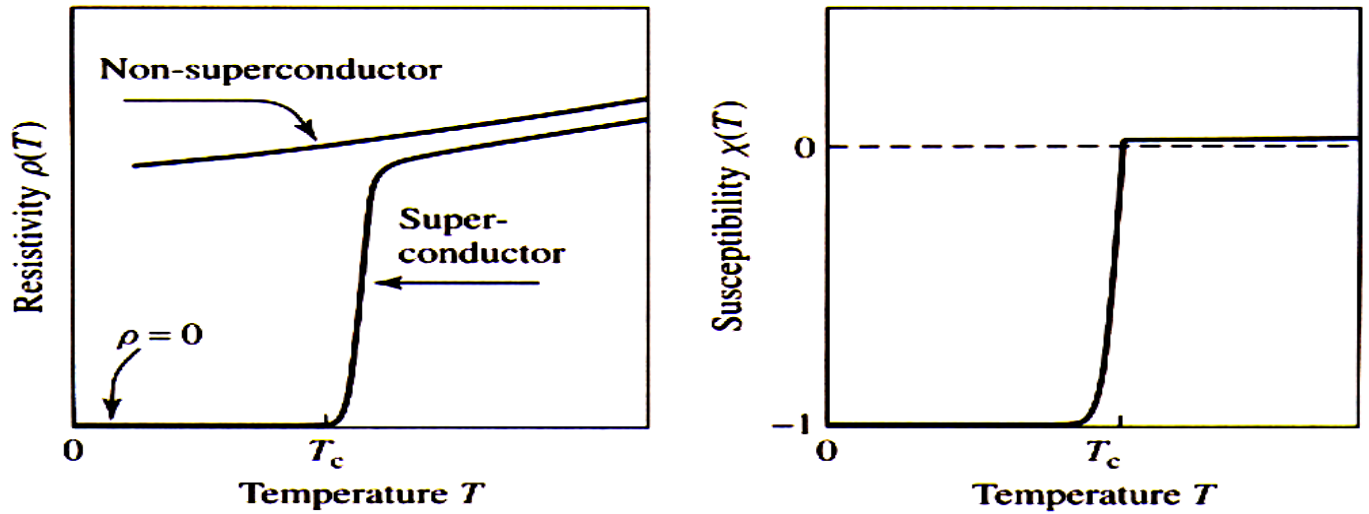


Figure-3(Resistivity vs temperature)

1.2.1 Zero electrical resistance:

Some materials or alloys show zero resistivity below some critical temperature which is called transition temperature. This is the property of a superconductor. Above this temperature these material show normal metallic behavior. This figure below shows the difference between superconductor and non-superconductors.

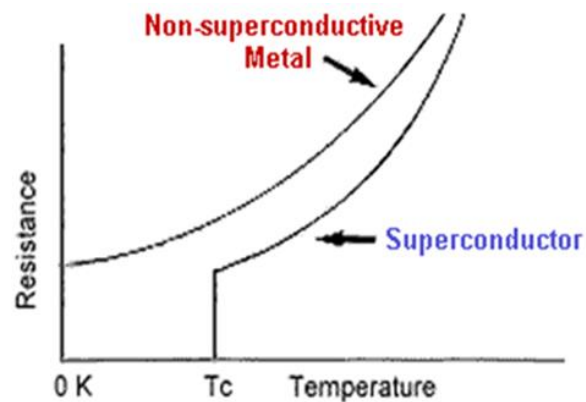


Figure-4 (electrical resistivity vs temperature)

1.2.2 Meissner Effect:

A superconductor having zero magnetic field inside it is said to be in the Meissner state. Meissner and Ochsenfeld measured magnetic flux arrangement outside superconducting specimens which has been cooled below their transition temperature while in a magnetic field. This specimen is also known as perfectly diamagnetic. Inside a superconductor, magnetic flux is zero.

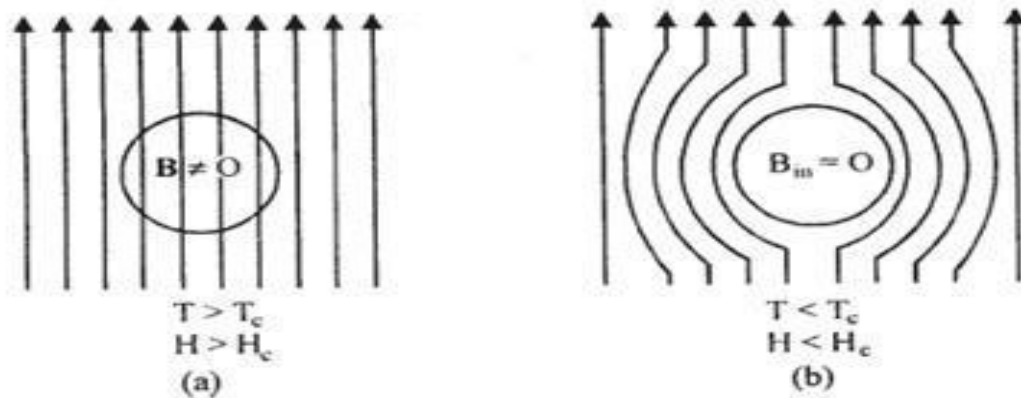


Figure-5

1.2.3 Effect of magnetic field:

The superconducting state of a metal exists only in a particular range of temperature and field strength. Superconducting state appear in the metal is that some combination of temperature and field strength should be less than critical value. It will disappear if the temperature of the specimen is raised above its T_c or sufficiently strong magnetic field is employed.

$$H_c = H_0 \left[1 - \left(\frac{T}{T_c} \right)^2 \right]$$

Where, T_c :Critical temperature the highest temperature of superconductivity.

H_c : Extreme critical field strength at the temperature T .

H_0 : Extreme critical field strength occurring at absolute zero

1.2.4 Effect of current:

The magnetic field which causes a superconductor to become normal state from a superconducting state is not necessarily by an external applied field. It may arise as a result of electric current which flows inside the conductor. The least current without disturbing its superconductivity that can be passed in a sample, that is called critical current I_c . If a wire of radius 'r' of a type-1 superconductor carries a current 'I', there is a surface magnetic field, $H_i = I/2\pi r$, which is associated with the current. If H_i becomes greater than H_c , the materials go normal state. If a transverse magnetic field 'H' is applied to the wire in addition to H_i , the condition for the normal state at the surface is that the sum of the applied field and the field due to the current should be equal to the critical field.

$$H_c = H_i + 2H$$

This is called Silsbee's rule. The critical current I_c will decrease linearly with increase of applied field until it reaches zero at $H = H_c/2$. If the applied field is zero, then $I_c = 2\pi r (H_c)$.

1.2.5 Penetration depth:

F.London and H.London described the Meissner effect and zero resistivity by adding the two condition electrical resistivity = 0 and magnetic field = 0 (from Meissner's effect) to Maxwell electromagnetic equation. According to that the applied field does not suddenly drop to zero at the surface of the superconductor, then decays exponentially according to the equation,

$$H = H_0 \exp\left(-\frac{x}{\lambda}\right),$$

Where H_0 - value of magnetic field at the surface

λ - Penetration depth; λ is the distance for H to fall from H_0 to H_0/e .

1.3 APPLICATION:

- Magnetic Levitation allows trains to “float” on strong superconducting magnets (MAGLEV in Japan, 1997). These work because a superconductor repels a magnetic field so a magnet will float above a superconductor. This virtually eliminates the friction between the train and the track. However, there are safety concerns about the strong magnetic fields used as these could be a risk to human health.

- To generate high Magnetic field e.g. for Magnetic Resonance Imaging (MRI).
- A SQUID (Superconducting Quantum Interference Device) is the most sensitive magnetometer. (sensitive to 100 billion times weaker than the Earth's magnetic field).
- Quantum Computing.
- Large hadron collider or particle accelerator. Superconductors are used to make extremely powerful electromagnets to accelerate charged particles very fast (to near the speed of light) [9].

CHAPTER-2

YBa₂Cu₃O_{7-δ}HTSC:

Superconductivity has wide applications in many fields including energy storage, electrical power transmission, high speed trains, large physics machines, optoelectronics magnetic levitation, magnetic shielding etc. so, there is always a demand for materials having superconducting properties, which also should be less expensive and better than the former one. Synthesizing a superconducting material having transition temperature close to room temperature is a major challenge for the scientists. Heading towards this path the current, focus of much of the research, development and commercialization of superconductors is on YBCO.

Yttrium barium copper oxide, often formulated as YBCO, is a crystalline chemical compound. This was the first material to become superconducting at about 92 K which is beyond 77 K, the boiling point of liquid nitrogen. The significance of the discovery of YBCO is the much lower cost of the refrigerant used to cool the material to below the critical temperature.

The dimensions of a single unit cell of YBCO are $a = 3.82\text{\AA}$, $b = 3.89\text{\AA}$ and $c = 11.68\text{\AA}$. The lattice is composed of so-called perovskite layers (ACuO_3) where A is a rare-earth or alkaline-earth element (e.g. or Ba in YBCO). The term $7-\delta$ in the chemical formula implies a slight shortage of oxygen. If $\delta = 0$, the lattice is in the orthorhombic phase whereas in the circumstance of $\delta = 1$, the material has a tetragonal structure and it will be a semiconductor.. Only the orthorhombic structure is superconducting but it is stable only at temperatures below 500°C .

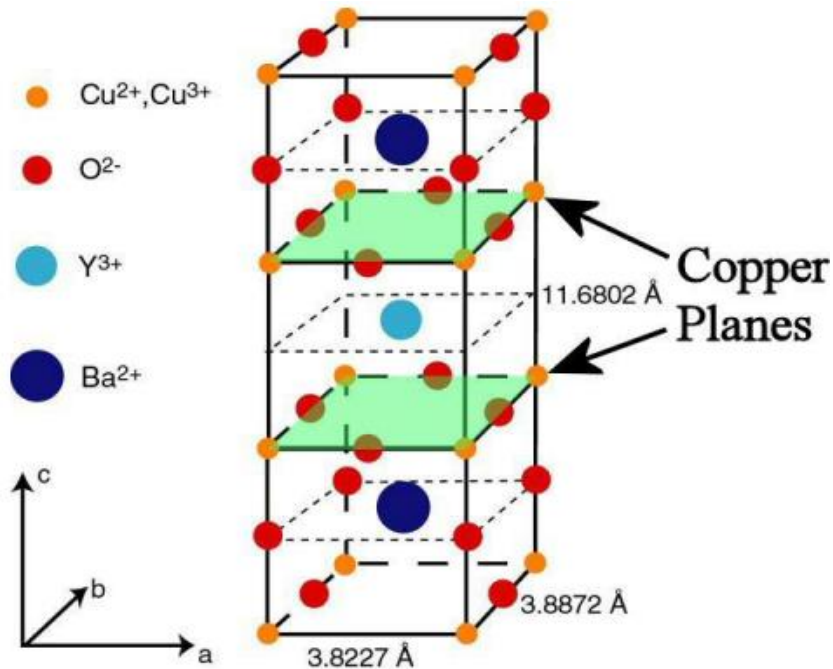


Figure-6 (Structure of a single unit cell of YBCO)

It has a penetration depth of 120 nm in the ab plane, 800 nm along the c axis and coherence length of 2 nm in the ab plane, 0.4 nm along the c axis. YBCO has a perovskite structure with layers and an oxygen deficiency. The boundary of each layer is defined by planes of square planar CuO_4 units sharing 4 vertices. Perpendicular to these CuO_2 planes is CuO_4 ribbons sharing 2 vertices. The yttrium atoms are found between the CuO_2 planes, while the barium atoms are found between the CuO_4 ribbons and the CuO_2 planes. Another important aspect of $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ material is that, like the other cuprates has an isotropic behavior, as a consequence of its crystalline structure that is reflected in the directional dependence of λ , ξ & H_{c2} [7]. The anisotropy of these parameters is remarkable between the c-direction & the a or b direction,

while the anisotropy between the a & b direction is small & can be neglected in most cases. The anisotropy of HTS materials can be described using Ginzburg- Landau theory that introduces a different effective mass of the hole carriers in different directions.

Carbon Nanotube:

CNT is a cylindrical form of carbon with diameter as small as 1nm. It is configurationally equivalent to a two dimensional graphene sheet rolled into a tube. These carbon molecules have unusual properties which are valuable for nanotechnology, electronics, optics and other fields of material science and technology. CNTs are allotropes of carbon with a cylindrical nanostructure. High temperature superconductivity denotes superconductivity in materials with high transition temperatures, accompanied by high critical current and magnetic fields [5]. But in YBCO, for magnetic impurity it has weak flux pinning for which critical current is low. So by pinning the flux lines artificially, one can prevent the vortex movement and increase the critical current density [4]. So we will prepare samples with more artificial pinning centers to increase critical current. Nano particles can increase critical current under high magnetic field [3].

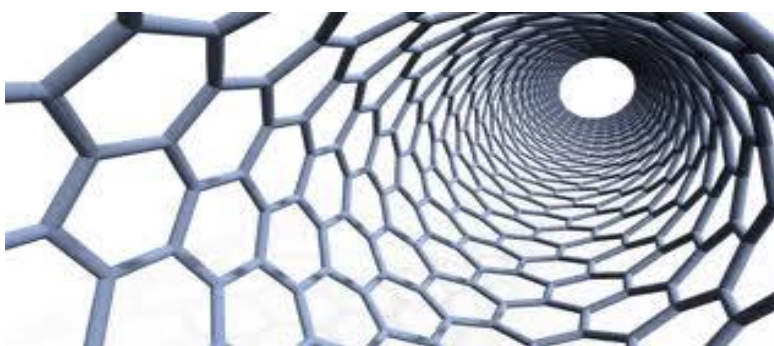


Figure-7 (CNT)

CHAPTER-3

SAMPLE PREPARATION:

3.1PREPARATION OF YBCO:

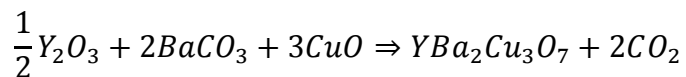
High temperature superconductors are prepared by a method called Solid state reaction method.

This method follows four steps:

- 1- Mixing of the powders
- 2- Calcinations at 920⁰ c
- 3- Intermediary grinding
- 4- Annealing in presence of oxygen at 500⁰ c.

This reaction takes place at high temperature. After the reaction has taken place, the YBCO should be reground to a powder and then pressed into a pellet.

YBCO was prepared by mixing the chemicals as per the balanced chemical equation.



Steps involved for the synthesis of this compound are given below:

1. Yttrium oxide, Barium Carbonate, Copper Oxide wastaken in stoichiometricratio (1:2:3) to prepare YBCO. The raw materials were measured using the high precision balance correct up to 4 decimal places.

2. The mixed sample was taken and was thoroughly ground in agate mortar for nearly 2 hour so as to obtain a mixed powder of the raw materials.
3. The ground sample was taken in an alumina crucible and calcined at 920°C for 12 hours for 4 times followed with 1 hour of grinding each time after the furnace was cooled.
4. The mixtures were taken out after the temperature reached room temperature.
5. Orthogonal structure was confirmed from XRD analysis.
6. For sintering the powdered sample was pressed in the form of cylindrical pellets of 0.5 grams each. The pelletization involves the uniaxial pressing using rigid dies with a pressure of 100MPa.
7. The YBCO pellets were sintered in a furnace at 900°C for 12 h, in normal atmosphere and 6 h in oxygen atmosphere.

Calcination:

After synthesis, the powdered sample is calcined in a tubular furnace for 12 hours at 900°C temperatures. The furnace takes nearly 4 hours and some minutes to reach the desired temperature after which the sample was calcined at the desired temperature for 12 hours. Then the furnace was cooled and the sample was thoroughly ground to ensure that mixing is proper and no agglomerates in the sample. The calcination temperatures are affected by the electrical and mechanical properties of the ceramics to a large extent. Calcination leads to the formation of the desired phase of the superconducting YBCO. This calcination decomposes the carbonates, nitrates or other impurity phases.

3.2 Preparation of YBCO/CNT composites:

High temperature preparation techniques such as arc discharge or laser ablation were first used to produce CNTs but nowadays these methods have been replaced by low temperature chemical vapor deposition (CVD) techniques ($<800^{\circ}\text{C}$), since the orientation, alignment, nanotube length, diameter, purity and density of CNTs can be precisely controlled in the CVD process. The most utilized methods and some of other non-standard techniques like liquid pyrolysis and bottom-up organic are preferred. Here, the multi walled CNT was prepared by liquid pyrolysis method.

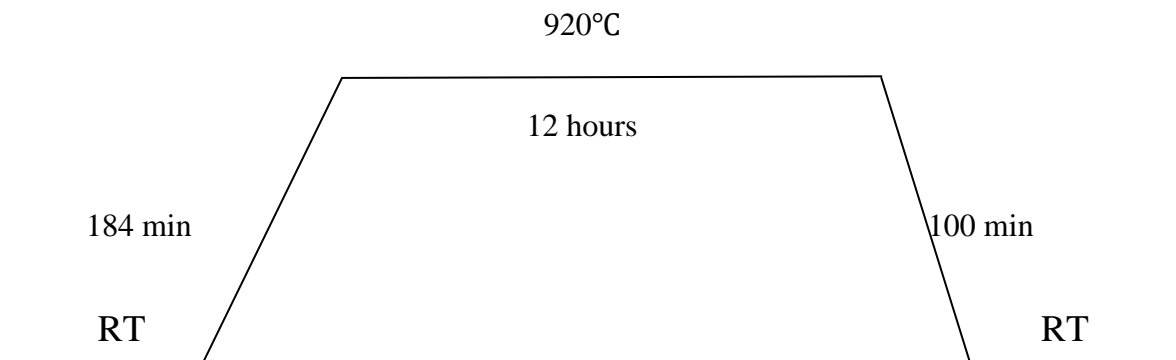
YBCO/CNT composites:

CNT was mixed with pure YBCO in fixed ratio. They are ground individually for one hour each and pressed in the form of pellets using pelletizer.

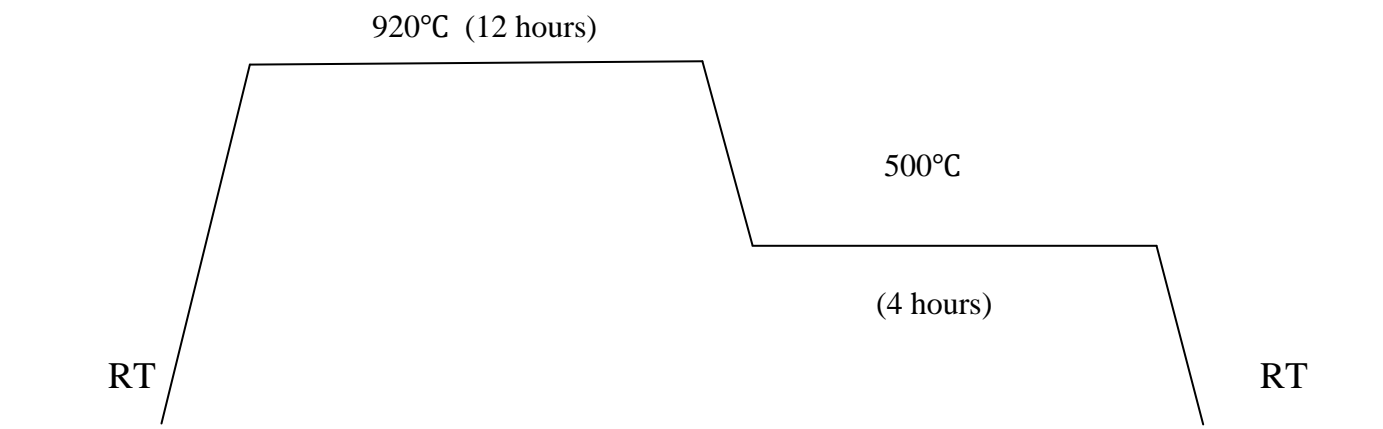
Sintering:

The powdered sample was pressed in the form of cylinder-shaped pellets of 0.5 grams each. The pellets were then sintered in a conventional furnace at 900°C for 12 hours and annealed at 500°C in the presence of oxygen. In the sintering stage the pellet shaped sample is heated to produce the desired microstructure by the reduction in grain boundary volume and increase in particle contact region.

Heating profile:

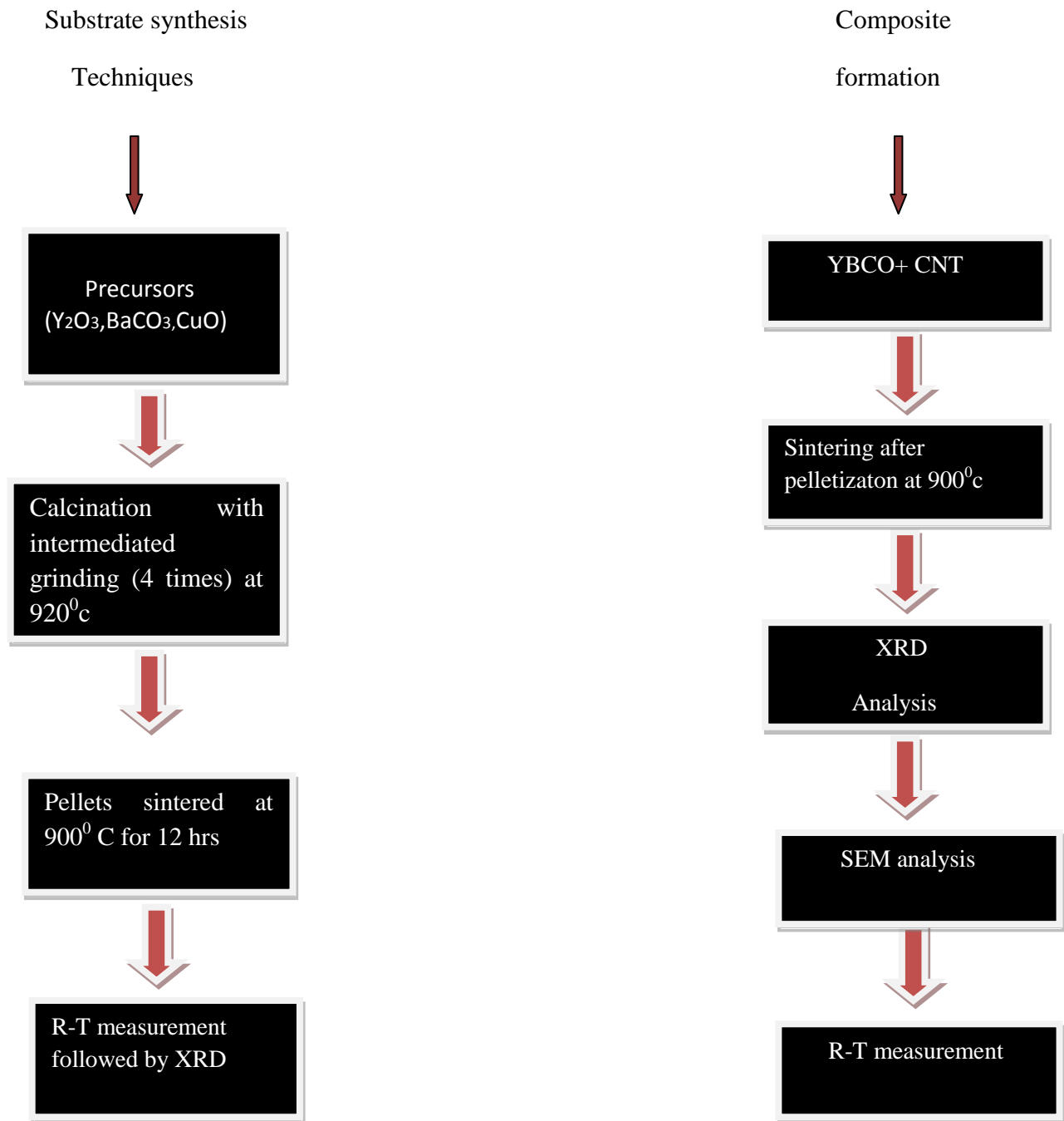


Heating profile for calcination



Heating profile for sintering

PREPARATION FLOW CHART:



CHAPTER-4

CHARACTERIZATION:

4.1X-RAY DIFFRACTION:

When an energetic electron beam is incident on a sample, then the core electron of the sample is ejected and the shell became vacant. To fill up this vacancy an excited electron from higher shell will come down to the core shell. Then an energetic ray will be emitted from that sample. This ray is called X-ray. This technique is based on the basic principle of Bragg's law which states that when a parallel monochromatic X-ray beam of wavelength ' λ ' incident at an angle of θ is diffracted by a set of parallel planes of a crystal (Fig.), then

$$2d\sin\theta = n\lambda$$

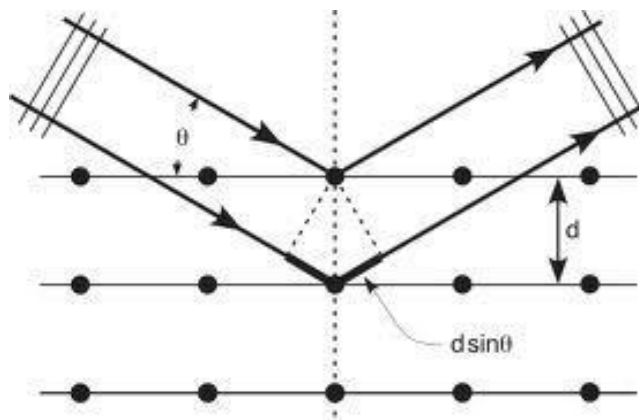


Figure-8 (Bragg's law)

4.2 Scanning Electron microscopy (SEM):

When an energetic beam of electrons is incident on a sample, the x rays, secondary electrons and back-scattered electrons, the auger electrons are ejected from the sample. The detector used in the SEM collects these electrons basically the secondary electrons and back scattered electrons and converted them into signal and displays on the SEM screen. The samples which are characterized by SEM are basically non-conducting, so a thin layer of silver is coated. SEM provides the information about the surface of the specimen because it cannot scan deeply into the surface. In SEM, small area of the sample can be analyzed.. This provides the information about the surface morphology, orientation and crystal structure of the sintered samples.

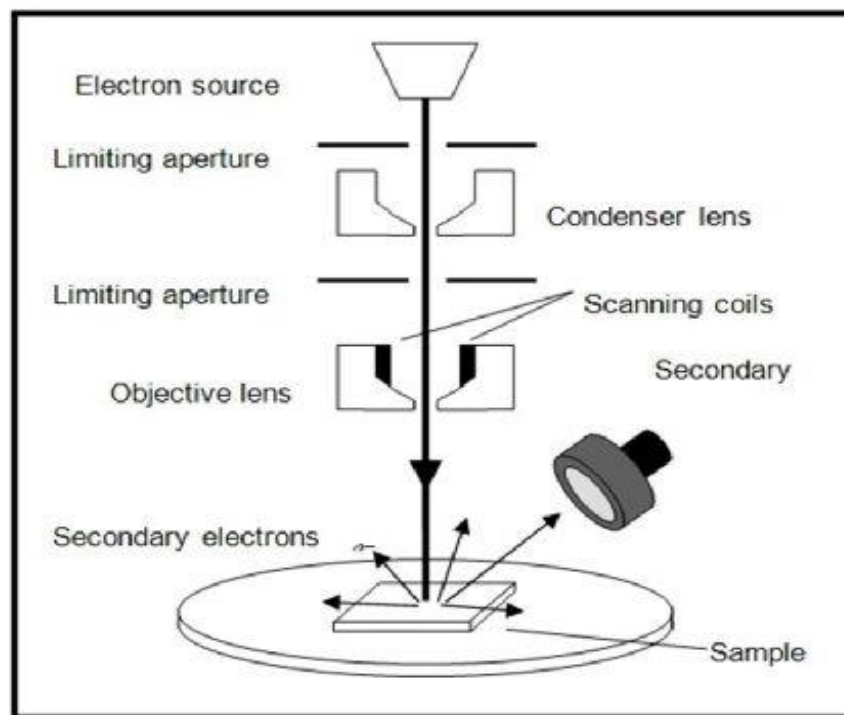


Figure-9 (SEM)

4.3 LOW TEMPERATURE R-T MEASUREMENT BY FOUR PROBE

METHOD:

Low-temperature physics, science concerned with the production and maintenance of temperatures much below normal, down to almost absolute zero, and with various phenomena that occur only at such temperatures. The temperature scale used in low-temperature physics is the Kelvin temperature scale, or absolute temperature scale, which is based on the behavior of an idealized gas. Low-temperature physics is also known as cryogenics, from the Greek meaning "producing cold." [8] Low temperatures are achieved by removing energy from a substance. This may be done in various ways. The simplest way to cool a substance is to bring it into contact with another substance that is already at a low temperature. Superconductivity and super fluidity have been thought of as phenomena that occur only at temperatures near absolute zero, but by the late 1980s several materials that exhibit superconductivity at temperatures exceeding 100°K had been found..

There is generally minority carrier injection by one the current carrying contacts. An excess concentration of minority carriers will affect the potential of other contacts and modify the resistance of the material.. It permits measurement of resistivity in samples having a wide variety of shapes, including the resistivity of small volumes within bigger pieces of semiconductor. In this manner the resistivity of both sides of p-n junction can be determined with good accuracy before the materials is cut into bars for making devices. This method of measurement is also applicable to silicon and other semiconductor materials. The true 4-wire measurement uses the current-potential method. The four points probe contains four thin collinear probes are made to

contact the sample under test. Current flow between to outer probes and voltage V_s is measured between the outer probes and voltage V is measured between the two inner probes, ideally without drawing any current. The resistance of the lead wires is not a factor because the value of the current is equal at any point in the circuit. It is self-regulating of the resistance of the lead wire and the input impedance of the voltage measurement is high enough to prevent any significant current flow in the voltage leads. Since no current is flowing, the voltage along the potential leads does not change along their length. The voltage drop is measured between the two probes voltmeter and current was measured by AC and DC current Source. Temperature Controller creates was used for temperature measurment.

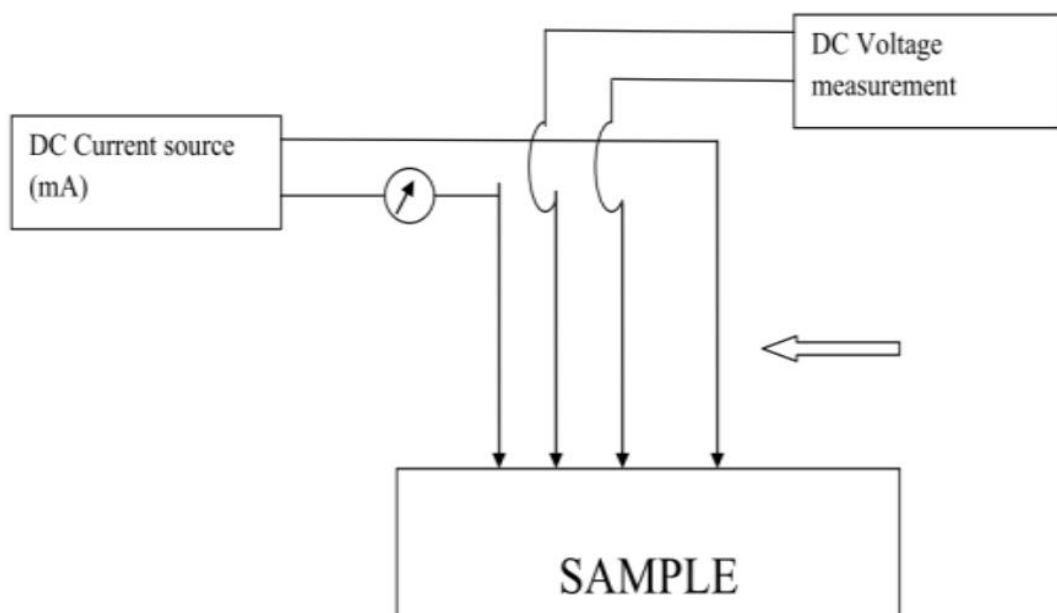


Figure-10 (Four probe method)

CHAPTER-5

RESULT AND DISCUSSION:

5.1XRD analysis:

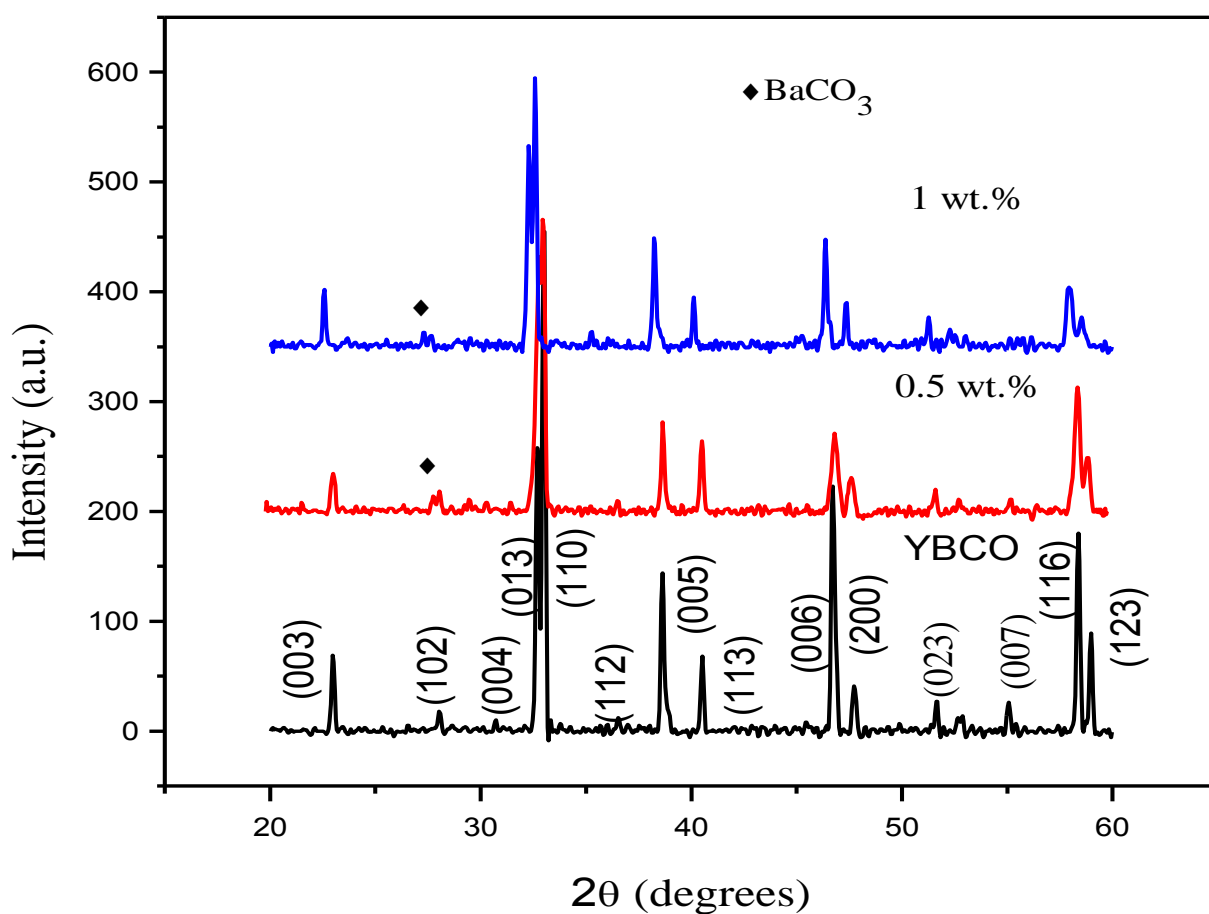


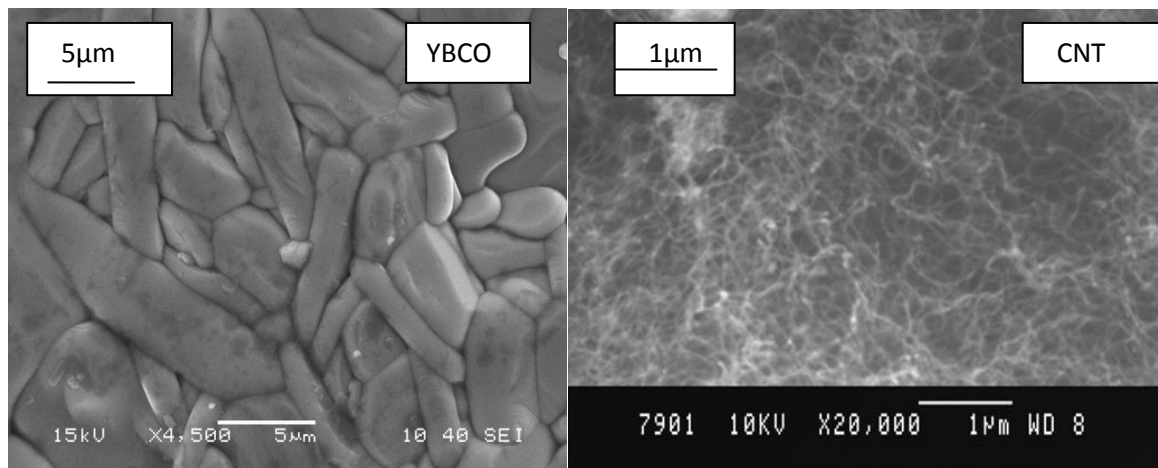
Figure-11

Fig.11 shows the diffraction pattern of the composite samples of YBCO/ x CNT(x = 0.0, 0.5, 1 wt.%) and were matched using Xpert High score software. Appearance of preferred orientation (00l) are shown with peaks indexed at (003), (004), (005), (006) and (007) in the XRD pattern of

YBCO .With CNT insertion in YBCO no peaks of CNT is detected. A secondary peak appears which grows within the parent superconducting phase. Analysis of the data reveals that the extra peak corresponds to BaCO_3 phase. With increasing of CNT content, broadening in the XRD peaks is shown prominent with decreased intensity.

5.2 SEM:

The morphological analysis was done by using scanning electron microscope. Fig.12 shows SEM images of the pure YBCO, CNT and YBCO/CNT composite. The microstructure characterization, i.e. grain boundary distribution in the superconductor composites is connected to electrical properties. The SEM picture in Fig. 12a shows that the pure YBCO has bad grain connections. Fig. 12(b) shows the pure CNT grains. When doped with CNT, it seems that the grain connection becomes a little better (Fig.12c). more close look with higher magnification reveals that the carbon nanotubes exist in between the grains and connect the grains electrically.



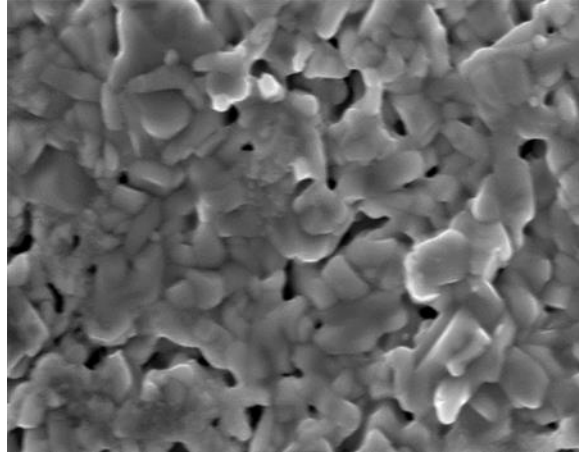
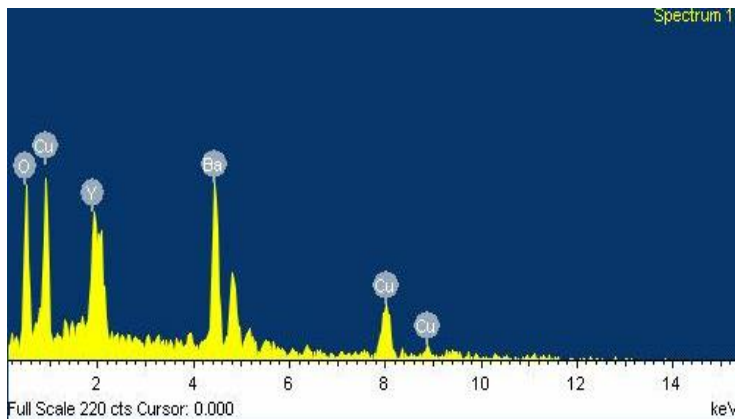


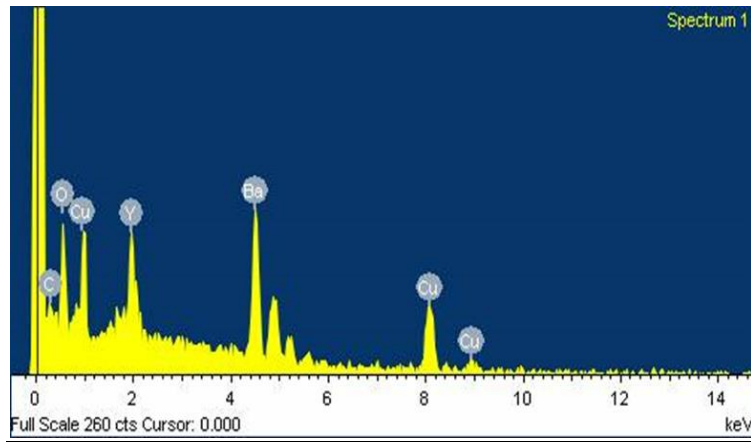
Fig. 12(a,b,c) . SEM micrographs of YBCO, CNT and YBCO+xCNT(x =1 wt.%) samples

5.3 EDS ANALYSIS:

The elemental analysis and their percentage composition were confirmed by EDS. The presence of the elements Yttrium, Copper, Barium, and Oxygen was observed by the peaks of EDS. The presence of Carbon was also confirmed by EDS image after CNT insertion. The weight % and atomic % of the composition is given in the tables below.



Element	Weight%	Atomic %
O	15.50	52.22
Cu	26.11	22.16
Y	12.65	7.67
Ba	45.74	17.95

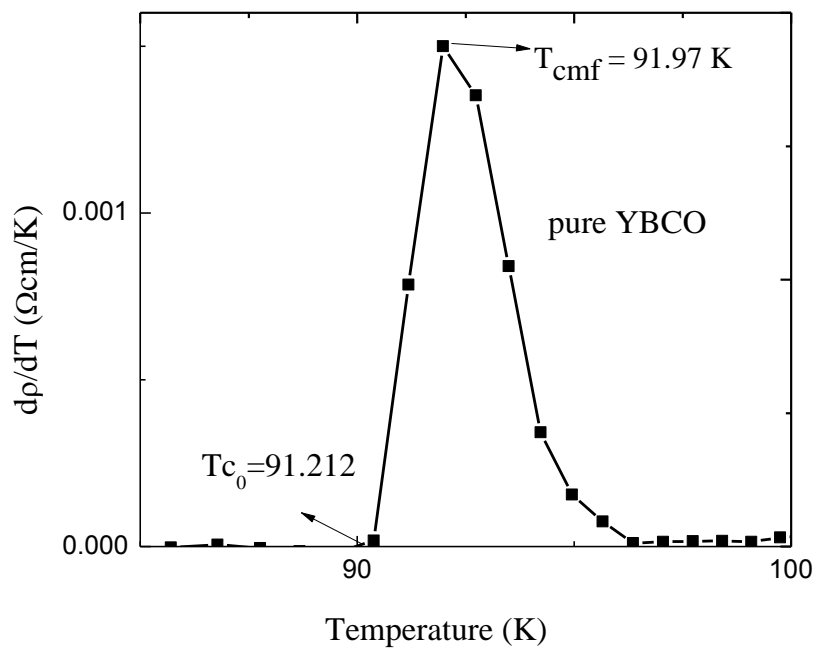
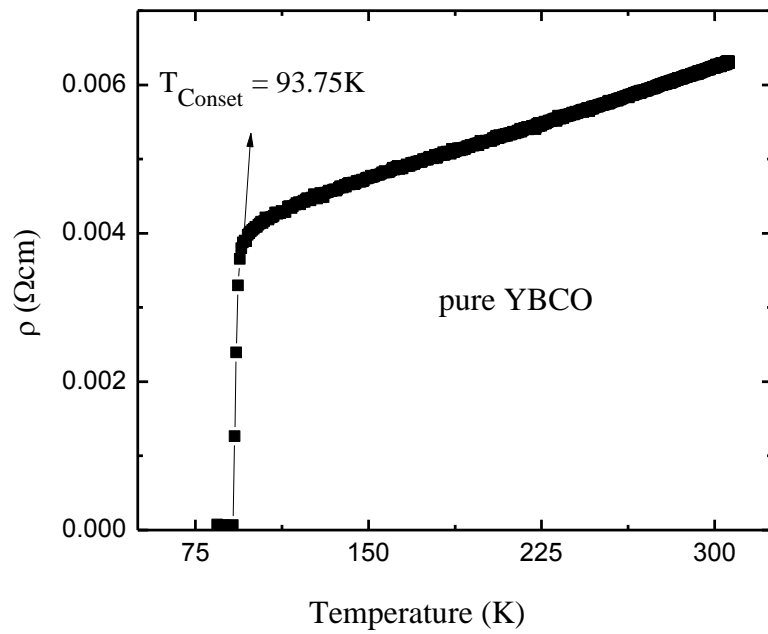


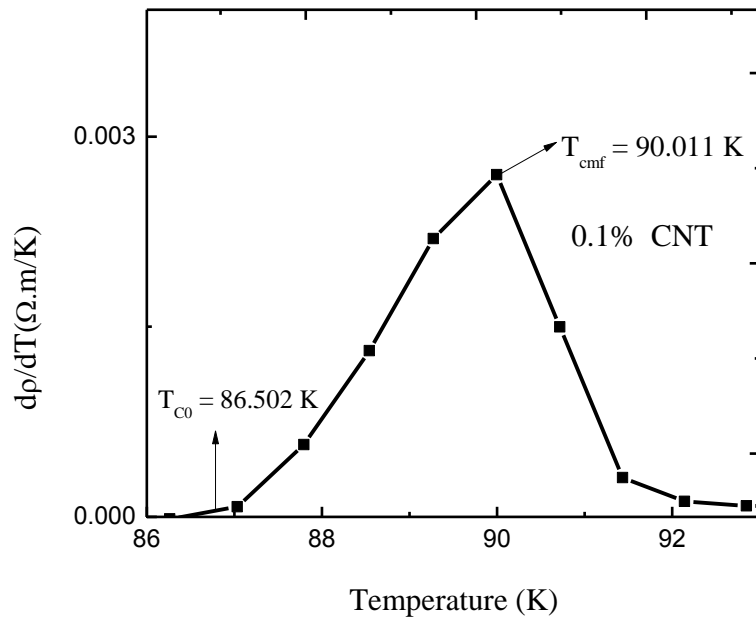
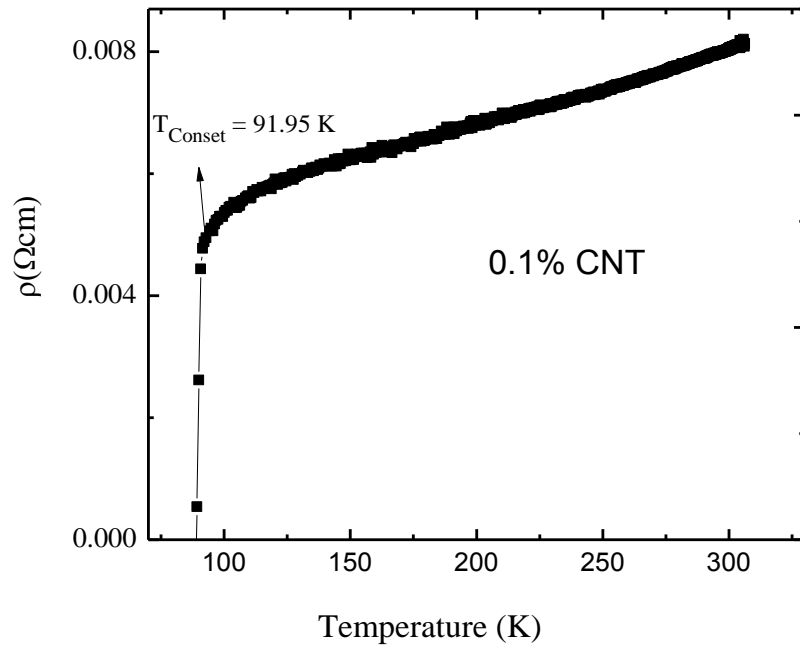
Element	Weight%	Atomic%
C	5.86	19.93
O	18.53	47.34
Cu	23.74	15.27
Y	12.49	5.74
Ba	39.38	11.71

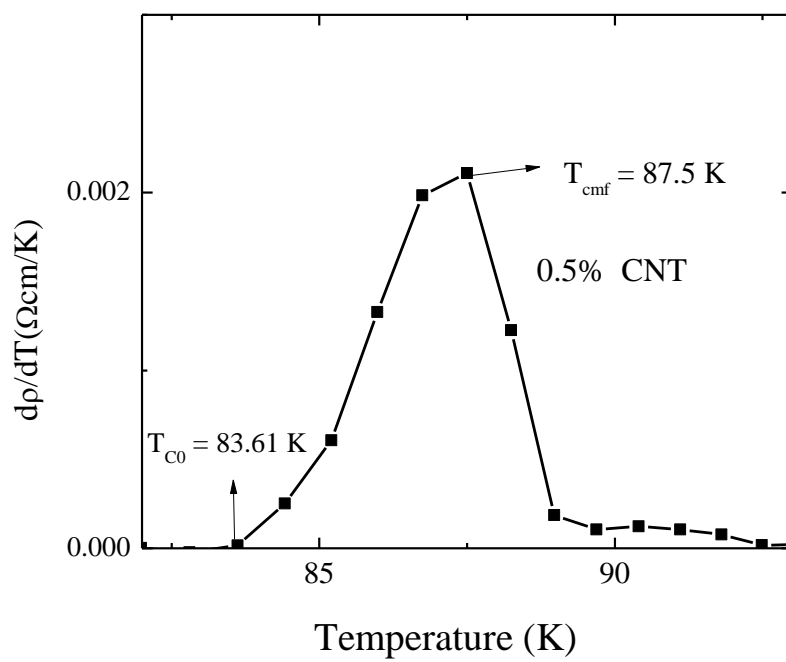
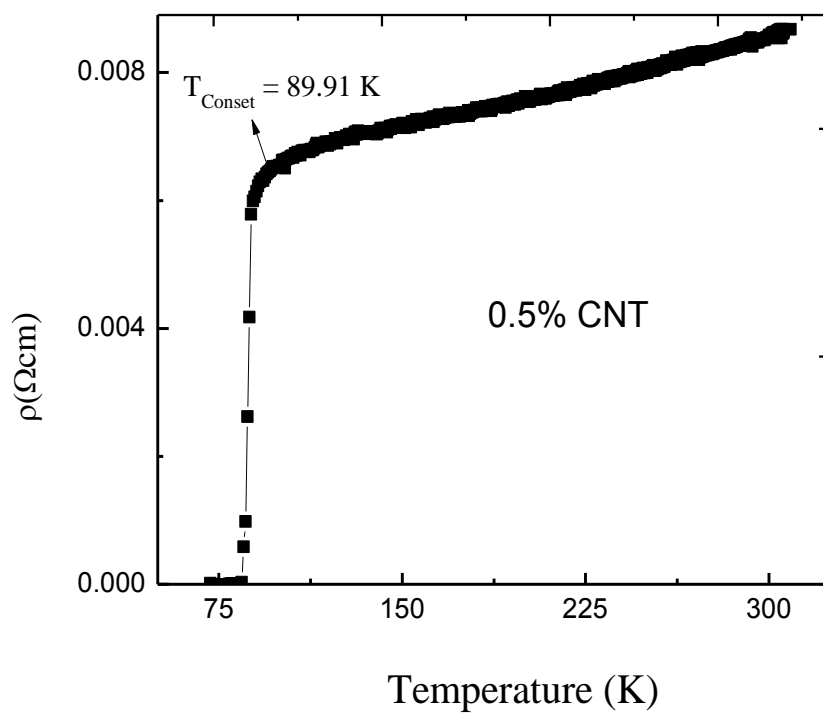
Fig. 13. EDS picture of YBCO+xCNT (x= 1 wt. %) samples.

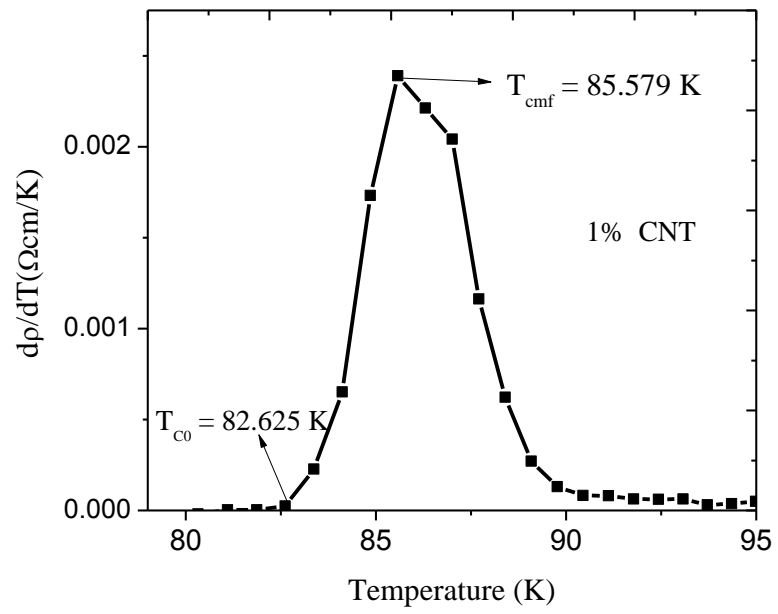
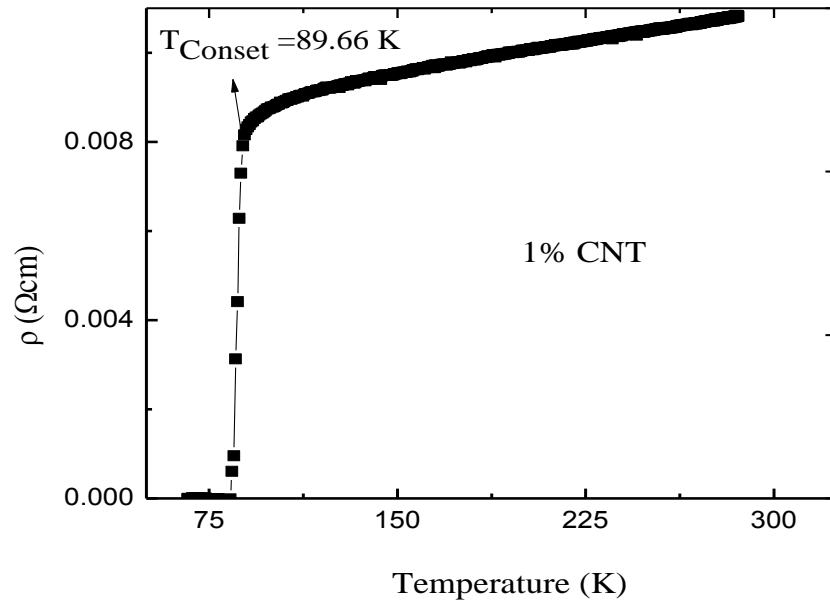
4.4 RESISTIVITY VS TEMPERATURE:

The resistivity dependence of temperature for different samples are measured with various amounts of YBCO+CNT and pure YBCO and are shown below. It is observed that $T_{c\text{onset}}$ is affected due to addition of CNT in YBCO. Metallic behavior is shown by all samples in the normal state and a superconducting transition to zero resistance. And all samples' properties depend linearly at high temperature. The resistive transition exhibits two different regions. The first region is characterized by the normal state which shows a proper metallic behavior. The second region is characterized by a superconducting stage which is due to the Cooper pair fluctuation to the conductivity below $T_{c\text{onset}}$, where resistivity is deviating from linearly with temperature. This is because of the increasing rate of fluctuation of Cooper pair on decreasing the temperature. And the temperature derivative plot of resistivity graph shows additional information about the superconducting parameters.









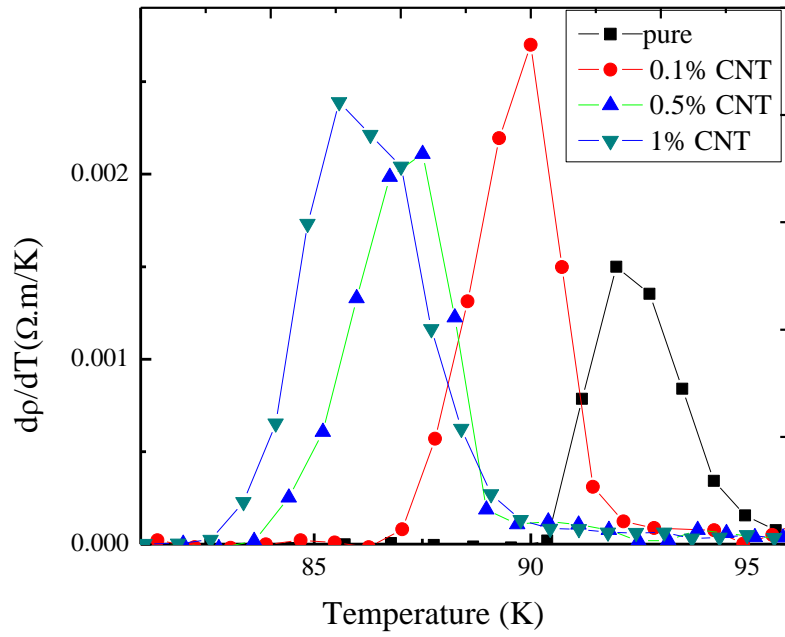
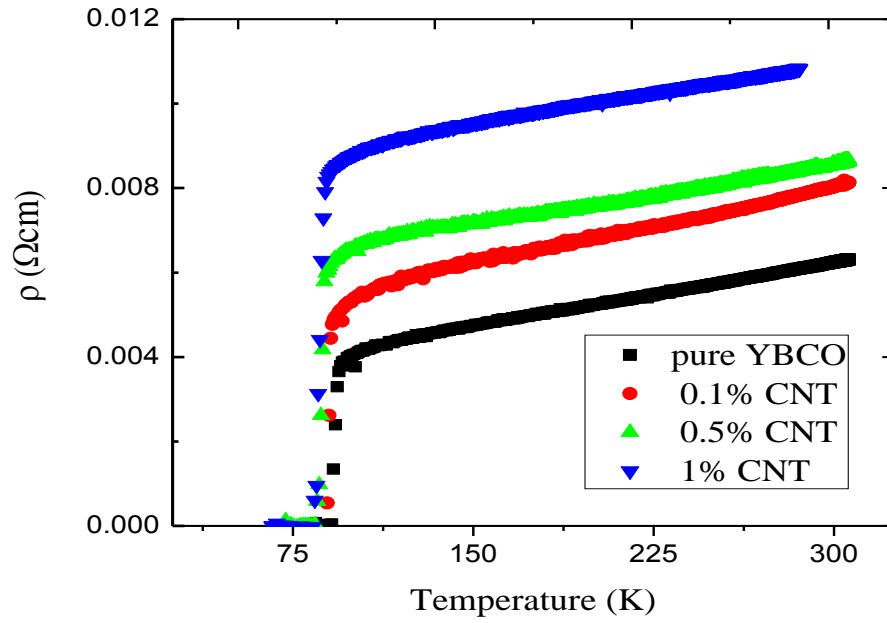


Fig.14 Temperature dependence of the resistivity for YBCO + x CNT($x = 0.0, 0.1, 0.5, 1$ wt. %) composites and their respective derivatives

From the peak of derivative plot one obtains the mean field transition temperature T_c , the temperature at which maximum slope change is occurring. $T_{c\text{onset}}$, is the temperature from which the sample starts to become superconductor whereas T_{c0} is the temperature at which the resistance completely becomes zero. The R-T plot shows no change in trend after low concentration of CNT addition to the matrix of the YBCO. However, we observe about 2 times increment of ρ (273) for 1% of CNT addition to pristine YBCO. The residual resistivity values also increases for non-metallic CNT insertion. The R-T data can be further analyzing by taking the derivative plot of the R-T data. The derivative plot highlights a peak that refers to maximum slope change indicating by T_{cmf} . We observe a shift of T_{cmf} towards the lower temperature zone with impurities addition. T_{cmf} recorded for YBCO pristine is 91.974K and decreases by 2K each for 0.1%, 0.5% and 1% CNT tabulated in table no.- Further the derivative plot shows zero resistivity i.e. total drop of resistance at T_{c0} . The T_{c0} values also decrease. The comparison of T_{c0} and T_{cmf} highlights that T_{c0} is highly affected than T_{cmf} . The concentration of CNT plays a crucial role in varying the different superconducting parameters T_{cmf} , $T_{c\text{onset}}$ and T_{c0} . Different critical temperatures are observed from the different composites.

Table for parameter:

Composition	$T_{c\text{onset}}$	T_{c0}	T_{cmf}	ρ_{273}	W(FWHM)	ΔT ($T_{c\text{onset}} - T_{c0}$)
Pure YBCO	93.750 K	91.212 K	91.974 K	0.0059	1.910	2.55
0.1% CNT	91.950 K	86.501 K	90.011 K	0.0076	2.04	5.44
0.5% CNT	89.90 K	83.610 K	87.502 K	0.0082	2.127	6.3
1% CNT	89.61 K	82.624 K	85.579 K	0.0107	2.735	6.98

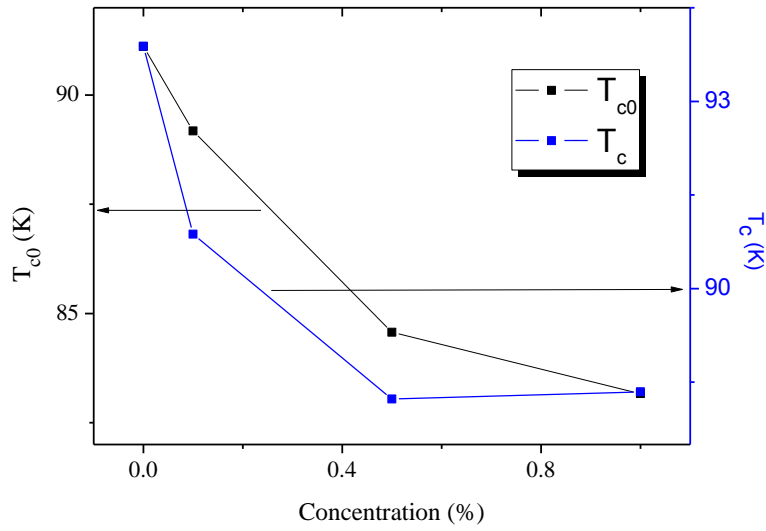


Figure 15: T_{c0} and T_c v/s wt. % CNT.

The various parameters are evaluated and compiled in table 1. Figure shows a plot of T_{c0} and T_c with different concentration of CNT. It shows the clear decreasing trend of T_{c0} and T_c with increasing weight of CNT. The grain boundaries are more affected by addition of CNT than the grain itself. The increase in ΔT and FWHM indicates that the transition temperature width increases. This may be attributed to the decrease in the phase coherence between the grain boundaries thereby increasing the grain boundary resistivity.

CHAPTER-6

CONCLUSION:

The YBCO/CNT composite is successfully synthesized by solid state reaction route. The role of well known CNT non superconducting inclusion in YBCO matrix is studied. XRD peaks reveal the formation of single phase perovskite structure for the composition. From SEM images the change in grain structure are studied, which occurred due to addition of CNT. From EDS data, the amount of carbon are found to known present in the composite. From R-T graph, it is concluded that with increase in wt. % of CNT in YBCO matrix, the critical temperature T_c and T_{c0} decreases gradually, which affects the electrical properties. Increasing value of ΔT signifies that CNT addition affects the intergranular links. And there is such a change in grain size with the addition of CNT. The pinning properties of CNT can be utilized for increment of critical current density of the composite system.

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